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# Refractory Properties of Brick Produced From Beneficiated Chrome-Containing Furnace Linings

By Arthur V. Petty, Jr.

UNITED STATES DEPARTMENT OF THE INTERIOR

I 28.23:

Report of Investigations 8685

# Refractory Properties of Brick Produced From Beneficiated Chrome-Containing Furnace Linings

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UNITED STATES DEPARTMENT OF THE INTERIOR
James G. Watt, Secretary

BUREAU OF MINES
Robert C. Horton, Director

Research at the Tuscaloosa Research Center is carried out under a memorandum of agreement between the Bureau of Mines, U.S. Department of the Interior, and the University of Alabama.

This publication has been cataloged as follows:

Petty, A. V

Refractory properties of brick produced from beneficiated chrome-containing furnace linings.

(Report of investigations / United States Department of the Interior, Bureau of Mines ; 8685)

Bibliography: p. 16.

1. Bricks–Thermal properties. 2. Refractory materials–Recycling. I. Title. II. Series: Report of investigations (United States. Bureau of Mines) ; 8685.

TN23.U43 [TP832.F5] 622s [620.1'42] 82-4461 AACR2

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# REFRACTORY PROPERTIES OF BRICK PRODUCED FROM BENEFICIATED CHROME-CONTAINING FURNACE LININGS

By Arthur V. Petty, Jr.[1]


## ABSTRACT

In Bureau of Mines research on recycling chrome-containing refractory wastes, used refractories from argon-oxygen decarburization and electric steelmaking furnaces were beneficiated. By means of dry magnetic separation on minus 6-mesh crushed wastes, a concentrate was produced. After minor adjustments to the particle size distribution, full-size refractory brick were dry-pressed by a commercial producer and fired to 1,450° and 1,730° C. Standard testing procedures were used to determine the cold-crushing strength, modulus of rupture at 25°, 1,300°, and 1,500° C, hot load, thermal shock resistance, thermal expansion, and slag resistance. Optical and electron microscopy were used to determine grain-bonding mechanisms, and energy-dispersive analysis of X-rays was used to determine elemental distribution. The test results indicate that the beneficiated material could be used to produce a refractory product suitable for moderate-temperature applications.


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## INTRODUCTION

North America has no significant reserves of chromite ore. At present, existing deposits would be used only if outside sources were unavailable. Most of the chromite ore used in the United States (97 percent) is imported from the Eastern Hemisphere; the main suppliers are the Soviet Union, the Republic of South Africa, the Philippines, and Turkey (8).[2]

Chromite ore, in combination with various quantities of magnesia, is employed in the manufacture of basic refractories. Refractories of this type are used as linings in steelmaking and copper-smelting furnaces, rotary cement calciners, and glassmaking tanks, with the bulk consumed by the copper and steel industries. Approximately 20 percent of the chromite used in the United States is for refractory purposes (9). In 1979, about 300,000 tons of chrome refractories were consumed in this country.

Magnesia-chromite (mag-chrome) refractories are engineered so that the relatively impure chromite grains react at their surfaces with the relatively clean magnesia to form grain-boundary spinels. This provides direct bonding for needed hot strength. During this process, any free silica and other minor impurities are reacted to form refractory compounds, and the formation of low-melting glasses that could soften during subsequent heating is thus avoided. Chemical and mineralogical balances are required. A concern with used brick is that glass formers (such as silica, alkali metals, and alkaline earths) may be retained from slags or exposed by transgranular crushing. Glass is evidenced by metallographic examination, by high, but insignificant, cold strength, and by low, but critical, hot strength.

In pursuit of the Bureau of Mines objective to maximize minerals recovery from secondary sources, the Tuscaloosa Research Center investigated techniques for reclaiming, reforming, and reusing chromite-containing waste refractories from copper and steelmaking furnaces. Earlier published reports (7, 12) indicated that both the copper and steelmaking refractory wastes could be beneficiated by means of standard mineral beneficiation techniques. Screening tests using copper smelter refractories showed that metallic copper contained in these refractories could be readily recovered; however, recycling of the beneficiated refractory grain was limited because of the minus 65-mesh grinding required to liberate the metallic copper. This fine-sized material would require briqueting, calcining, and crushing to prepare a refractory grain coarse enough for reuse in the manufacture of mag-chrome refractories.

Work with both argon-oxygen decarburization (AOD) and electric arc steel furnace refractories, on the other hand, indicated that contaminants could be adequately removed from a minus 6-mesh material, yielding a concentrate of suitable particle size for direct recycling into refractories.

Based upon this earlier work, approximately 1,000 pounds each of waste AOD and electric steel furnace refractories were beneficiated by means of a combination of crushing, screening, and dry magnetic separation. With only minor adjustments to particle size distribution required (13), full-size refractory brick were dry-pressed by a commercial producer and fired to 1,450° and 1,730° C. This report presents the results of tests made to evaluate the refractory properties of these brick.

[2]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

ACKNOWLEDGMENTS

The author thanks Republic Steel Corp. for providing samples of used refractories and Harbison-Walker Refractories for manufacturing the full-size recycled refractory brick used in the investigations.

## BENEFICIATION STUDIES

Samples of waste mag-chrome refractories from AOD and electric steelmaking operations in Ohio were obtained for use in the study. No estimate was provided by the steel company as to the quantity of materials it generated or stockpiled. However, in 1974, the steel industry consumed 4.9 pounds of chromic oxide ($Cr_2O_3$) --as chrome-bearing refractories--per ton of steel produced (10).

The samples of waste refractories as shown in figure 1 consisted of whole as well as pieces of used refractory brick. Some of these brick were partially enclosed in metal casings; others had metal and slag readily visible in cracks; and some were contaminant free.

The waste refractories were crushed, screened, and magnetically separated as outlined in the flowsheet presented in figure 2. The metal casings from handpicking and the magnetic product from the magnetic pulley were combined. This combined product accounted for 1.3 and 4.5 percent of the total weights received for the AOD and electric waste refractories, respectively.

After the metal casings and magnetic contaminant were removed, the chemical analyses made of the nonmagnetic product or chrome concentrate were comparable to those of the commercial brick, as shown in table 1.

TABLE 1. - Chemical analysis of chrome concentrates and commercial brick, percent

| Sample | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | $Cr_2O_3$ | CaO | MgO |
|---|---|---|---|---|---|---|
| AOD furnace..... | 3.1 | 6.5 | 10.8 | 16.3 | 3.0 | 59.8 |
| Electric furnace..... | 3.0 | 10.8 | 8.8 | 15.0 | 5.6 | 56.7 |
| Commercial brick..... | 1.5 | 13.2 | 6.3 | 14.5 | .7 | 63.8 |

The material was crushed to pass 6 mesh, the size range used by industry in the manufacture of refractory brick. Chemical analyses were made of the various minus 6-mesh size fractions of each refractory waste sample to determine the percent of $Cr_2O_3$ in each fraction. The size analyses, $Cr_2O_3$ content, and $Cr_2O_3$ distribution in the various size fractions are shown in table 2. While there is some variation in the $Cr_2O_3$ content of the various size fractions, none was low enough in grade to be rejected as a tailings product.

TABLE 2. - Screen analyses of minus 6-mesh waste refractories, percent

| Screen size, Tyler mesh | Weight | | $Cr_2O_3$ analysis | | $Cr_2O_3$ distribution | |
|---|---|---|---|---|---|---|
| | AOD furnace lining | Electric furnace lining | AOD furnace lining | Electric furnace lining | AOD furnace lining | Electric furnace lining |
| Plus 10 | 19.5 | 29.5 | 16.0 | 11.0 | 19.4 | 21.9 |
| Minus 10 plus 14 | 23.3 | 20.2 | 16.2 | 13.9 | 23.8 | 19.2 |
| Minus 14 plus 20 | 13.0 | 12.0 | 16.3 | 16.3 | 13.1 | 13.7 |
| Minus 20 plus 28 | 10.2 | 9.5 | 16.9 | 19.1 | 10.6 | 12.3 |
| Minus 28 plus 35 | 5.9 | 5.7 | 17.1 | 20.2 | 6.3 | 8.2 |
| Minus 35 plus 48 | 5.4 | 5.3 | 17.0 | 19.6 | 5.6 | 6.9 |
| Minus 48 plus 65 | 3.9 | 3.4 | 16.4 | 18.3 | 3.7 | 4.1 |
| Minus 65 plus 100 | 3.8 | 3.1 | 15.8 | 16.7 | 3.7 | 3.4 |
| Minus 100 plus 150 | 3.1 | 2.6 | 15.5 | 14.5 | 3.1 | 2.7 |
| Minus 150 plus 200 | 2.6 | 2.3 | 15.0 | 12.9 | 2.5 | 2.1 |
| Minus 200 plus 270 | 1.6 | 1.4 | 14.7 | 11.7 | 1.3 | 1.4 |
| Minus 270 plus 400 | 2.4 | 1.8 | 14.9 | 11.7 | 1.9 | 1.4 |
| Minus 400 | 5.3 | 3.2 | 15.6 | 11.6 | 5.0 | 2.7 |
| Composite | 100.0 | 100.0 | 16.0 | 14.6 | 100.0 | 100.0 |

When chemical analyses of the beneficiated refractory wastes were compared with those of the commercial refractories, minor differences were observed in silica ($SiO_2$), alumina ($Al_2O_3$), and calcium oxide (CaO) (table 3). The slightly higher $SiO_2$ and lower $Al_2O_3$ contents in the refractory wastes were judged to be insignificant. An analysis of the CaO revealed no "free" lime; therefore, the presence of calcium silicates was likely. Although the presence of calcium silicates can pose a problem in the production of refractories because of hydration, no further beneficiation efforts were made.

TABLE 3. - Chemical analysis of beneficiated waste refractories and commercial refractories, weight-percent

| Sample origin | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | $Cr_2O_3$ | CaO | MgO |
|---|---|---|---|---|---|---|
| AOD furnace: | | | | | | |
| Minus 6, plus 10 mesh | 3.77 | 9.22 | 12.0 | 17.8 | 2.65 | 53.9 |
| Minus 10, plus 30 mesh | 3.70 | 9.23 | 12.3 | 18.8 | 2.53 | 52.8 |
| Minus 30 mesh | 3.87 | 9.34 | 12.2 | 18.6 | 2.86 | 52.3 |
| Electric furnace: | | | | | | |
| Minus 6, plus 10 mesh | 2.65 | 9.54 | 8.19 | 11.0 | 3.76 | 64.3 |
| Minus 10, plus 30 mesh | 2.71 | 13.4 | 9.07 | 17.0 | 3.95 | 54.0 |
| Minus 30 mesh | 3.39 | 12.0 | 9.20 | 14.2 | 8.41 | 51.9 |
| Commercial brick: | | | | | | |
| No. 1 | 1.5 | 13.2 | 6.3 | 14.5 | .7 | 63.8 |
| No. 2 | 2.0 | 17.9 | 9.4 | 20.7 | .6 | 49.4 |

Energy-dispersive analysis of X-rays showed only Mg, Cr, Al, Fe, Si, and Ca as the major elemental constituents of the samples. However, traces of Mo, S, V, and Ti were also observed in the AOD furnace sample, and Ti and Zn were observed in the electric furnace sample. Semiquantitative spectrographic analyses indicated that trace amounts of Ag, Co, and Ni were also present.

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FIGURE 1. - Waste mag-chrome brick from steel furnace lining.

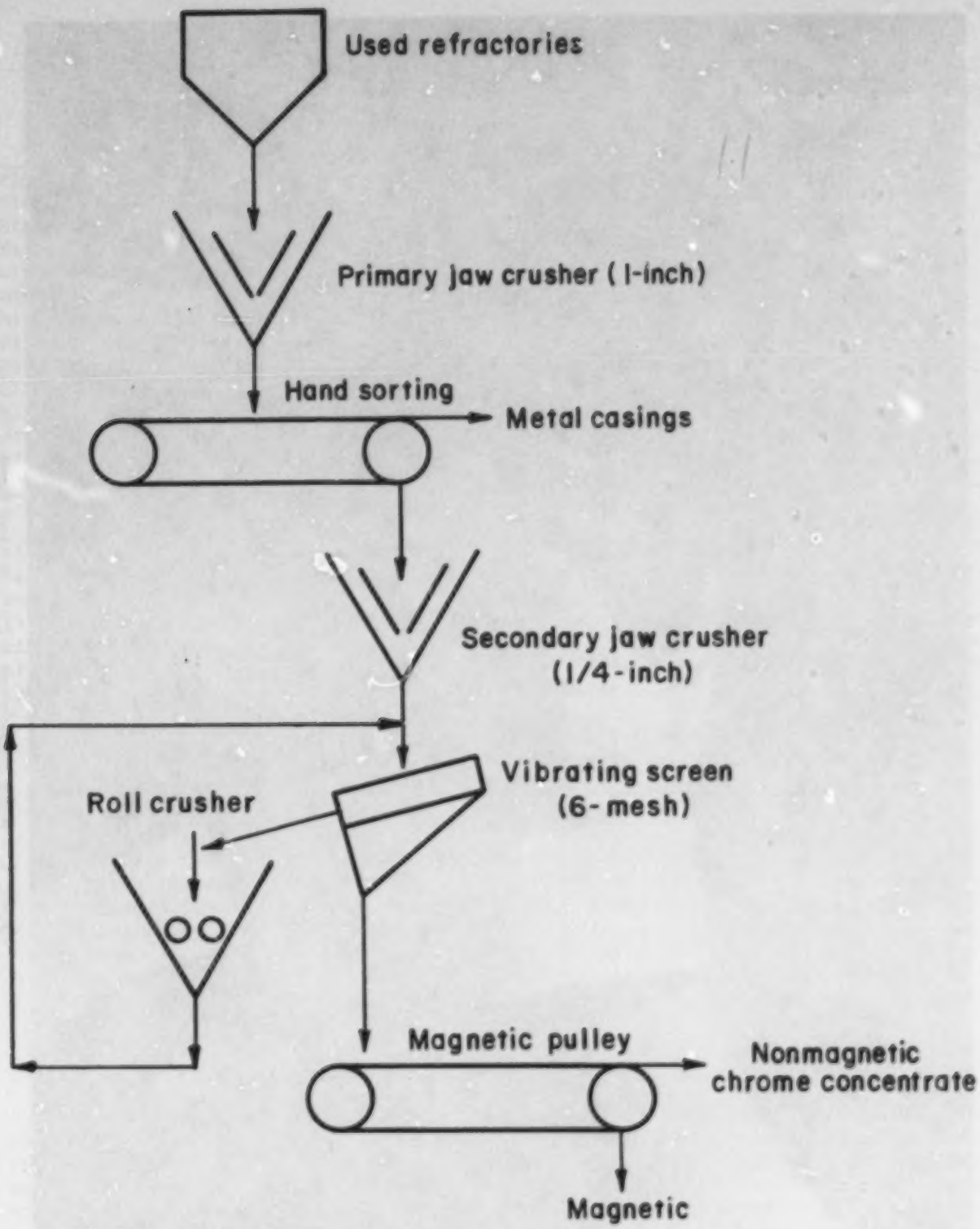



FIGURE 2. - Flowsheet for beneficiating waste refractories.

X-ray diffraction analyses were made of each beneficiated sample for mineralogical determinations. X-ray diffraction patterns were also valuable in determining that no significant phase changes had occurred in the refractory grain during furnace operations, which might alter the refractory properties of the beneficiated refractory grain (chrome concentrate).

Chromite ([Fe, Mg]O [Cr, Al, Fe]$_2O_3$), periclase (MgO), and minor amounts of forsterite ($Mg_2SiO_4$) were identified in both the AOD and electric furnace samples.

## REFRACTORY EVALUATION

The beneficiated samples of AOD and electric steel furnace refractories were screened and divided into three size fractions, minus 6- plus 10-mesh, minus 10- plus 30-mesh, and minus 30-mesh. The material was then shipped to a commercial refractory manufacturer for producing full-size brick. Portions of the three size fractions were recombined and suitable binders added to allow pressing of approximately sixty 9- by 4-1/2- by 3-inch brick.

No difficulty was encountered in pressing and burning the brick produced from beneficiated AOD linings. However, because of the high calcium content of the beneficiated electric arc furnace lining, the brick produced from this material hydrated and cracked during drying as shown in figure 3.

As shown in table 3, the bulk of the calcium present in the electric arc furnace refractories was found in the minus 30-mesh size fraction. In order to reduce the total calcium content, a portion of the minus 10- plus 30-mesh material was ground to minus 30 mesh, and additional bricks were pressed, using this instead of the original minus 30-mesh material. These brick showed much less severe cracking during drying, although small cracks were still evident. All bricks were pressed at 18,000 psi and fired to 1,450° C with a 10-hour hold at the maximum temperature.

Figure 4 shows examples of the brick produced from the recycled AOD and electric arc furnace linings. The magnified view of the surface of the electric arc furnace brick shows the hydration cracks present. Figure 5 shows composite photomicrographs of each of the recycled brick along with similar micrographs of a commercial mag-chrome refractory. These photographs show the presence of chrome spinels; periclase; sharp, angular secondary spinels, which have precipitated during cooling from a liquid phase; magnesioferrite; and silicates. It should be noted that large amounts of the glassy silicate phase are present at the grain boundaries for AOD brick fired to 1,450° C.

The bulk density and apparent porosity were obtained using ASTM C67-78 (3) and are presented in table 4. Similar data, supplied by the manufacturers, are given for the two commercial refractories. This table shows that the values obtained for the recycled bricks are equivalent to those obtained on commercial refractories of similar type.

TABLE 4. - Bulk density and apparent porosity for recycled and commercial brick

| Brick | Bulk density, g/cm$^3$ | Apparent porosity, pct |
|---|---|---|
| Recycled: | | |
| AOD..... | 3.19 | 15.3 |
| Electric..... | 2.97 | 19.7 |
| Commercial: | | |
| No. 1..... | 3.00-3.08 | 15.0-18.0 |
| No. 2..... | 3.06-3.14 | 16.0-19.0 |

Because of the hydration cracks present in the bricks produced from electric steel furnace refractories, they were

FIGURE 3. - Cracks resulting from hydration of electric arc furnace refractories during drying.

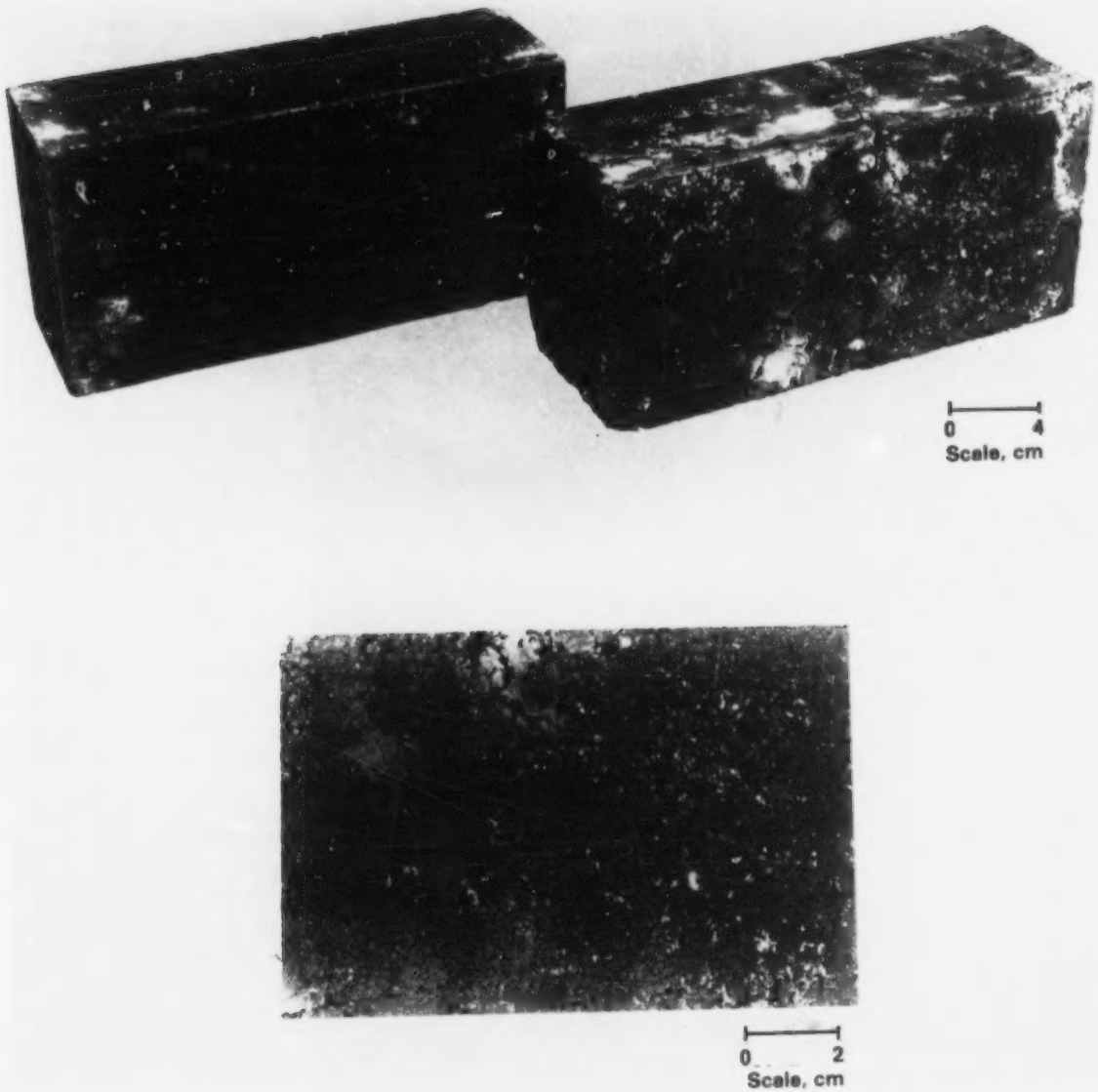



FIGURE 4. - Brick produced from recycled AOD (top left) and electric arc furnace (top right) refractory linings. Lower center shows magnified view of electric arc furnace brick surface with hydration cracks.

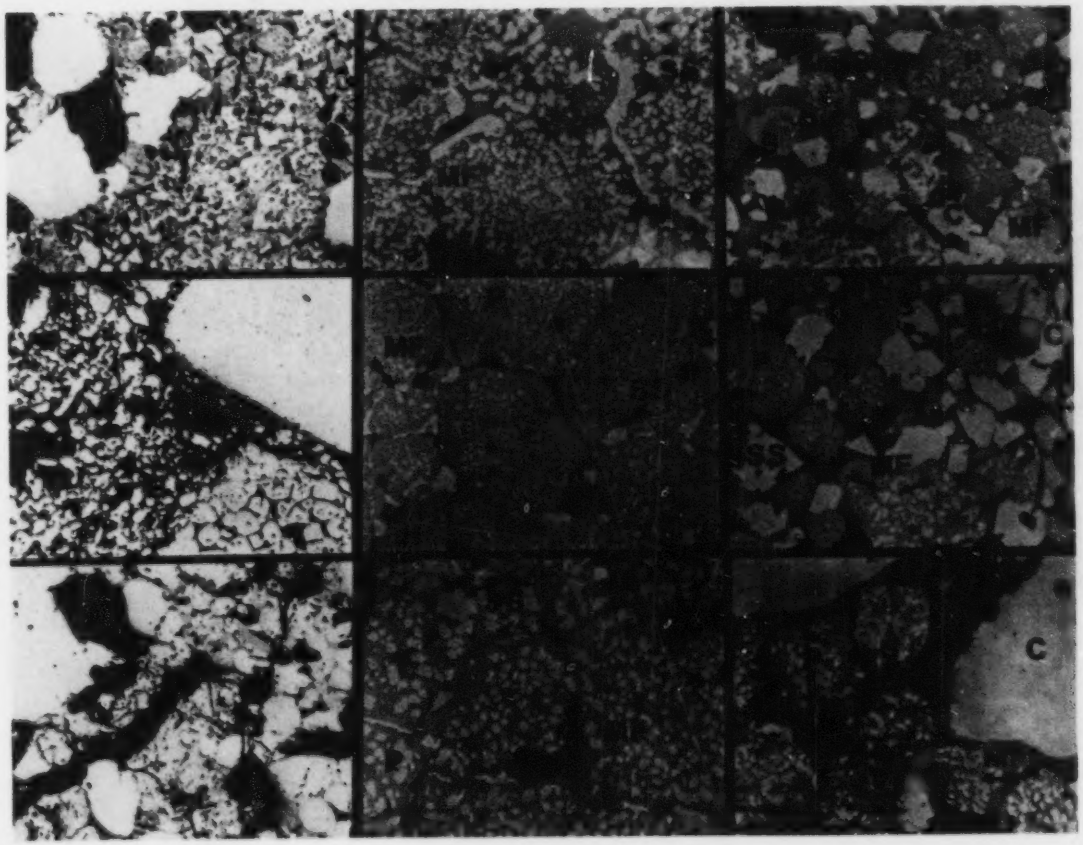



FIGURE 5. - Micrographs of recycled AOD and electric arc furnace bricks and commercial mag-chrome refractory. Designations are as follows: C = chrome spinel, P = periclase, SS = secondary spinel, MF = magnesioferrite, and S = silicate. The middle column shows concentrated areas of large-grained periclase with precipitated magnesioferrites within the grains and secondary spinels at the grain boundaries. The right column shows areas of smaller periclase and chrome spinel grains with associated secondary phases.

not subjected to further refractory evaluation. Samples of the recycled AOD refractory brick were prepared for additional testing of their refractory properties, including cold crushing strength, modulus of rupture at 25°, 1,300°, and 1,500° C, hot load, thermal shock resistance, and thermal expansion.

The cold crushing strength was determined using ASTM C133-72 (5). Five specimens 9 by 4-1/2 by 3 inches were tested, and an average value of 11,800 psi was obtained.

Values for modulus of rupture at ambient temperature were obtained, again using ASTM C133-72. Five 9- by 4-1/2-by 3-inch samples were evaluated, and an average value of 1,960 psi was obtained. Modulus of rupture at elevated temperatures was obtained using ASTM C583-76 (4). Five specimens, 1- by 1- by 9-inch and cut from each of five full-size brick, were tested at each of two temperatures. The average values for specimens evaluated at 1,300° and 1,500° C were 1,260 and 150 psi, respectively.

Hot load deformation was measured according to ASTM C16-70 (2). Two 9- by 4-1/2- by 3-inch specimens were subjected to a 25-psi load while being heated from ambient to 1,750° C over an 8-hour period (Schedule 5, Test to Failure). Average deformation for the two samples was minus 6.2 percent.

Thermal shock resistance was determined using the Bethlehem prism spalling test (1). Samples 2 by 2 by 3 inches were cycled (15 minutes heating at 1,200° C and 10 minutes air cooling) until each piece broke to yield a piece(s) at least one-third of the sample. The failure cycle was recorded for each piece, and the average is reported as the spalling resistance index. For the AOD recycled refractory, a value of 7.4 was obtained.

Thermal expansion was determined using a recording dilatometer and 3-inch sample. A heating rate of 60° C per hour, in air, was used. The data developed are shown in graphical form in figure 6. Over the temperature range evaluated, an almost linear curve was generated giving a value of $\alpha = 10.11 \times 10^{-6}$ in/(in·° C).

These data are summarized in table 5 along with manufacturers' data for two commercial refractories. It is noted that the cold crushing strength for the recycled refractory is twice that reported for the commercial samples, while the modulus of rupture is only about 25 percent.

TABLE 5. - Refractory properties of recycled waste AOD refractory lining and commercial brick of similar composition

| Test[1] | Beneficiated AOD lining | Commercial brick |
|---|---|---|
| Apparent porosity.....pct.. | 15.4 | 16.0-19.0 |
| Bulk density.....g/cm$^3$.. | 3.2 | 3.1 |
| Cold crushing strength.....psi.. | 12,000 | 4,000-6,000 |
| Modulus of rupture.....psi.. | [2]150 | [3]600-1,200 |
| Load test at 25 psi (deformation).....pct.. | [4]1.55 | ([5]) |
| Spalling resistance index..... | 7.4 | NA |
| Thermal expansion.....in/(in·° C).. | [6]10.11 x $10^{-6}$ | NA |
| Slag resistance (area loss).....cm$^2$.. | 3.0 | 3.64 |

NA Not available.
[1]All tests were according to ASTM specifications.
[2]At 1,500° C.
[3]At 1,480° C.
[4]For brick fired to 1,730° C.
[5]Negligible deformation under load to 1,800° C.
[6]For brick fired to 1,450° C.

The relatively low modulus of rupture value obtained for the recycled brick at 1,500° C and the corresponding high percent change measured by the load test at 1,750° C are due to the relatively low initial firing temperature these brick received. When refractories of this type are fired, a glassy-bond phase is present between the more refractory periclase and chrome-spinel grains

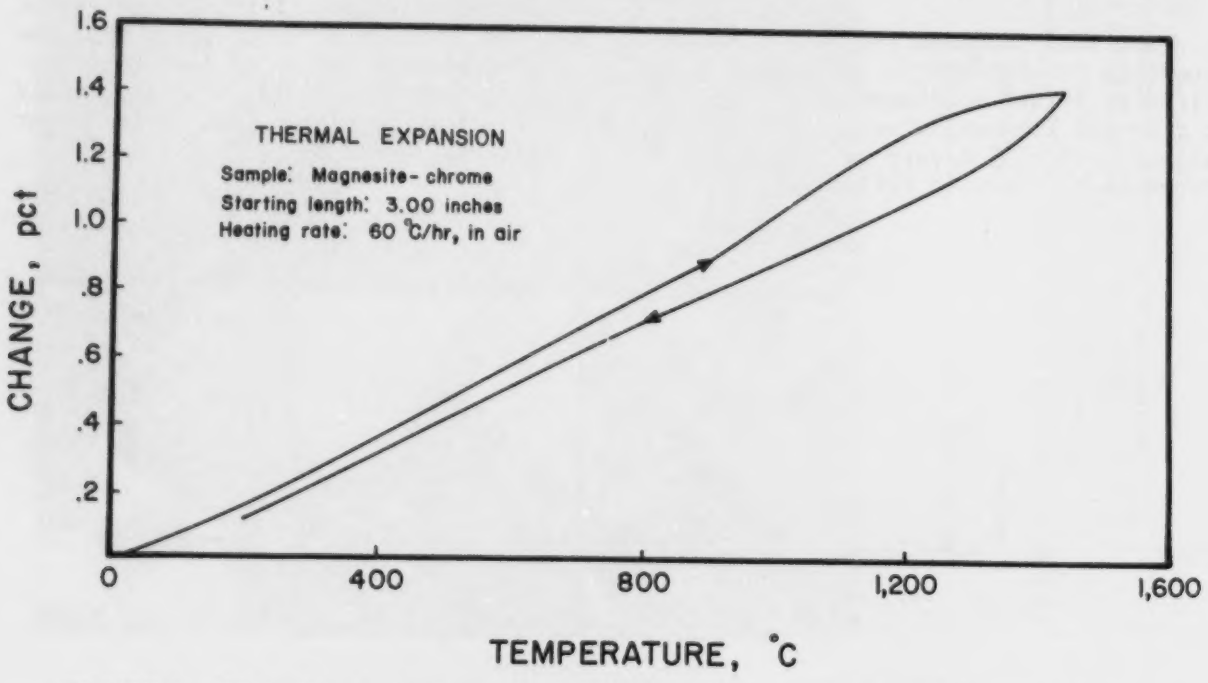



FIGURE 6. - Thermal expansion curve for brick produced from recycled AOD refractory lining.

up to at least 1,500° C. When refractories are fired to higher temperatures (>1,700° C), a direct bond is formed between the periclase and chrome-spinel grains, accompanied by a substantial decrease in the amount of glassy phase present at the grain boundaries. This decrease results from a redistribution of the silica, which reacts with magnesia to form forsterite and with calcium to form dicalcium silicate, both of which have high melting points.

In order to verify the formation of a direct bond, several brick previously fired to 1,450° C were refired to 1,730° C. Figure 7 shows the microstructures of samples after firing to 1,450° and 1,730° C. The direct bonding of periclase and chrome-spinel grains and the corresponding decrease in the amount of glassy phase present at the grain boundaries are apparent in the lower photograph. Hot load tests were repeated for brick after firing to 1,730° C, and deformation averaged only minus 1.55 percent, compared with the minus 6.20 percent reported for samples fired to 1,450° C.

The slag resistance of recycled AOD refractories was evaluated using a rotary slag-test apparatus developed at the Bureau of Mines, Tuscaloosa Research Center (6, 11). A schematic diagram of the apparatus used is shown in figure 8. Tests were conducted using a highly reactive basic oxygen furnace slag having the composition shown in table 6. The test was run for 8 hours (an initial 2-hour heat-up followed by 6 hours at 1,550° C) with 200-gram slag additions made at 10-minute intervals for a total slag throughput of 8.4 kilograms. Commercial refractory brick No. 2 was included in the test for comparative purposes.

TABLE 6. - Chemical analysis of slag used in rotary slag test, weight-percent

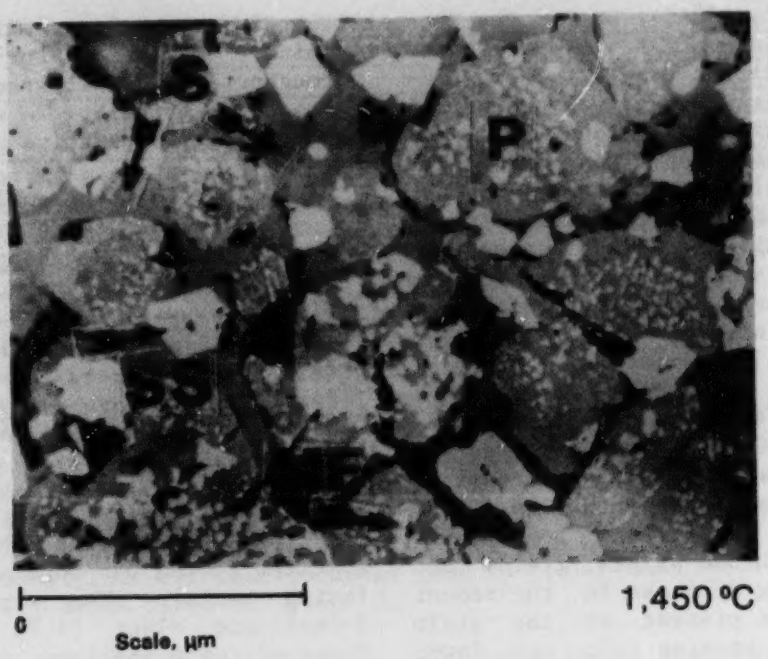
| | |
|---|---|
| $SiO_2$ | 33 |
| CaO | 33 |
| $Fe_2O_3$ | 20 |
| $Al_2O_3$ | 4 |
| MgO | 5 |
| MnO | 5 |
| Total | 100 |

The average area change for each specimen tested was obtained in the following manner: The straight (4.5- by 9-inch) and slant (4.7- by 9-inch) side faces of each specimen were traced on a piece of white posterboard before and after testing to represent the initial and final areas of the faces. Photocopies of these posterboards were made, and the area of paper representing the difference between the initial area and final area was cut out and weighed. The decrease in area for each face was determined from the weight of paper. The decrease in area for each specimen is the average of the area change for the two side faces. Average area change for the recycled AOD refractory was 3.00 centimeters, compared with 3.64 centimeters for the commercial refractory. The smaller change for the recycled brick would indicate superior resistance to slag erosion and corrosion.

## SUMMARY AND CONCLUSIONS

By using a combination of conventional beneficiation techniques such as crushing, screening, and dry magnetic separation, it was possible to beneficiate waste AOD and electric steel furnace mag-chrome refractories. Contaminants were liberated by crushing the waste refractory to minus 6 mesh, yielding a product having a particle size distribution suitable for recycling into secondary refractory products.

Refractories produced from recycled AOD linings compared favorably with conventional commercial mag-chrome refractories of similar composition. Cold



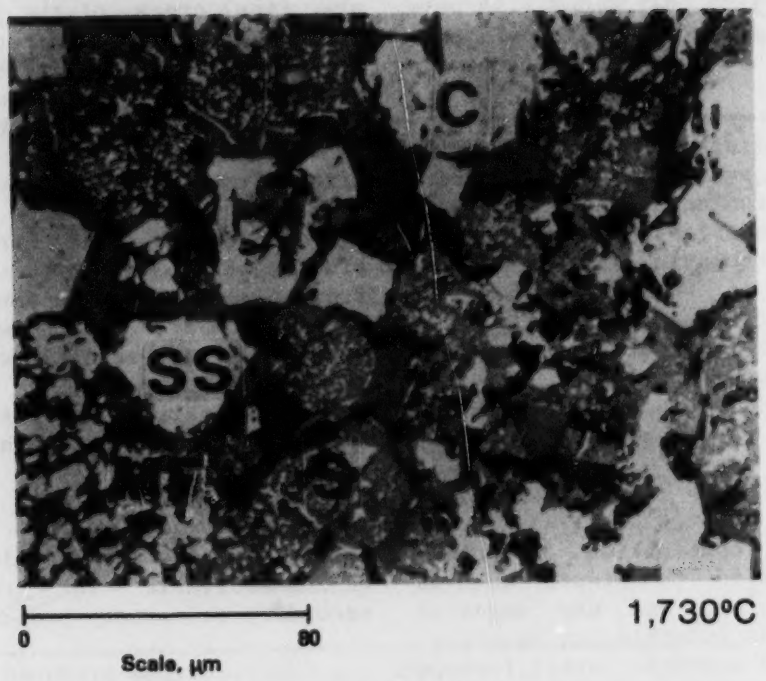




FIGURE 7. - Micrographs of recycled AOD refractory after firing to 1,450° and 1,730° C. Designations are as follows: C = chrome spinel, P = periclase, SS = secondary spinel, MF = magnesioferrite, and S = silicate.

crushing strength, modulus of rupture up to at least 1,300° C, and slag resistance were actually superior for the recycled brick. Firing the brick produced from beneficiated AOD refractories to temperatures of >1,700° C resulted in a direct-bonded brick with improved hot strength over that of brick fired to 1,450° C that had a glassy silicate-bond phase.

Refractories produced from recycled electric arc furnace linings developed cracks during drying, which resulted from the hydration of calcium silicates. Since the calcium content of the material was concentrated in the minus 30-mesh fraction, it could probably be reduced to acceptable levels through further beneficiation, although this would increase the cost.

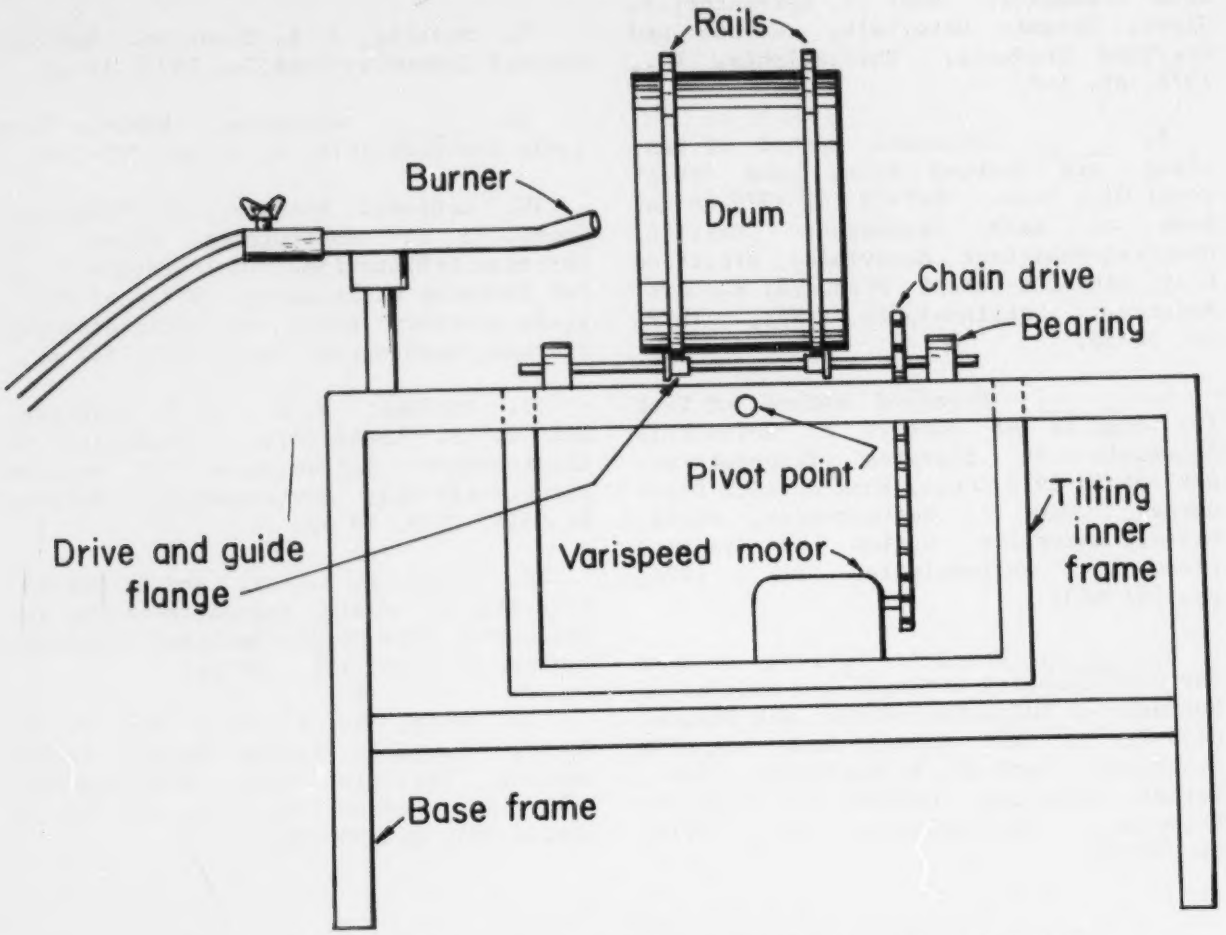



FIGURE 8. - Schematic of dynamic slag-testing apparatus and auxiliary equipment.

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INT.-BU.OF MINES,PGH.,PA. 26188